Advanced Surface Engineering Division
Room Ballroom A - Session SE-ThP

Advanced Surface Engineering Poster Session

SE-ThP-1 Can a Nanoindenter be Used as a Hardness Spectrometer?, ***Esteban Broitman,*** SKF B.V. - Research & Technology Development, Netherlands

The precise knowledge of material microstructures is of vital importance to understand their mechanical and tribological performance. Standard microstructural characterization, carried out by optical and electron microscopy together with X-ray diffraction, is usually correlated to the hardness determined by Rockwell or Vickers indentation at macro- and microscale, and nanoindentation at nanoscale.

In the first part of the presentation, the background of a novel statistical nanoindentation technique to measure hardness and Young's modulus of materials is described [1]. We indicate how experiments are designed, and how the distribution of the hardness and modulus of elasticity determined from the nanoindentation observations are deconvoluted to generate hardness histograms that reflect unique characteristics (fingerprints) of each coating or bulk material. We show how the statistical deconvolution analyses gives an estimate of the microstructural constituents, their volume fraction and corresponding plastic and elastic properties at nanoscale. In the second part, numerous examples on different kind of coatings and bulk materials are presented to illustrate the usefulness of the novel technique.

We demonstrate that, by using nanoindentation as a novel tool for static nanomechanical spectrometric analysis of coatings and bulk materials, a fundamental understanding of the relation between local microstructure (phases and their size) and local material response during elastic and plastic deformation can be obtained.

[1] *"Microstructural Analysis of Bearing Steels by a Statistical Nanoindentation Technique,"* E. Broitman, M. Y. Sherif, B. Minov, U. Sachadel, Bearing World Journal 5 (2020) 47-54.

SE-ThP-2 Diagnosing Stress in Thin Films with High-Throughput Experimentation and Simulation-Based Methods, ***Matias Kalaswad,*** *A. Shrivastava, S. Desai, J. Custer, S. Addamane, M. Rodriguez, P. Kotula, M. D'Elia, H. Najm, R. Dingreville, B. Boyce, D. Adams,* Sandia National Laboratories

Stress in metal thin films is a critical aspect of fabrication processes, as excess residual stress can have a detrimental effect on the reliability, performance, and durability of devices. Although there are well-established methods to determine the amount of residual stresses in thin films, unraveling all sources of stress remains challenging due to the numerous factors involved in thin film deposition. Similarly, efforts to quantify relationships between film microstructure and stress remain challenging because of the multimodal nature of microstructural data. Here we seek to identify material "fingerprints" utilizing high-throughput characterization together with simulations to identify process-structure-property correlations related to the development of stress in metal thin films. Mo thin films were deposited onto oxidized Si wafers by magnetron sputtering over various Ar pressures and sputter powers. Traditional wafer curvature measurements were first performed to obtain average residual stress results consistent with those reported in the literature. An automated, high-throughput XRD process was then used to determine out-of-plane lattice spacings in agreement with the in-plane stress according to the Poisson effect. In-plane images of the film were collected through scanning electron microscopy (SEM) and atomic force microscopy (AFM) complemented by measurements of film resistivity. To supplement the experimental results and add to the multi-fidelity of information, simulations of metal transport were completed to estimate the deposition profile and rate as well as properties of the metallic flux during sputtering.The Monte Carlo code SIMTRA, simulating the transport of atoms from the source to the substrate during PVD, provides estimates of ion energy distributions and angular distributions of arriving species, and these characteristics were then applied to a phase-field model to produce the growth dynamics of microstructures resembling those acquired experimentally (verified by transmission electron microscopy). The synthetic and experimental information was ingested by machine learning models with architectures adept at identifying patterns in multimodal data streams. Initial attempts are made to identify meaningful process-structure-property relationships from the experimental and simulated data using a multimodal-based deep learning model. The deep learning model will be further analyzed to explore the dominant mechanisms that underlie the stress developed in metal thin films.

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SE-ThP-3 Significant Texture and Wear Resistance Improvement of TiN Coatings Using Pulsed DC Magnetron Sputtering, ***Nicholas Richter,*** *B. Yang, J. Barnard, T. Niu,* Purdue University; *Y. Zhang,* Los Alamos National Laboratory; *D. Shaw,* Advanced Energy Industries, Inc.; *H. Wang, X. Zhang,* Purdue University

Titanium nitride (TiN) coatings fabricated through reactive sputtering feature a suite of parameters capable of altering the microstructure and properties. Here, we explore the influence of using a bipolar pulsed direct current (DC) power supply in place of conventional DC when depositing TiN coatings, focusing on the influence of pulse frequency. The implementation of a pulsed DC voltage profile promotes a drastic texture change from randomly oriented to strongly TiN(111) across the full range of pulse frequencies. Additionally, higher pulse frequencies promote significant grain size reduction, which directly influences the resulting mechanical properties. Nanoindentation and nanoscratch testing were conducted and revealed a corresponding increase in hardness and wear resistance, respectively. The mechanism for this microstructural and texture change is also explored.

SE-ThP-4 Investigation of Laser Ablation Coating Removal (LACR) for Steel Surface Cleaning and Coating Adhesion, ***William Moffat,*** University of Virginia; *J. Provines, S. Sharp,* Virginia Transportation Research Council (VTRC); *S. Agnew, J. Fitz-Gerald,* University of Virginia

Laser cleaning has recently received much attention for its ability to clean and prepare metallic surfaces for recoating and bonding operations such as welding, mold cleaning, surface texturing, paint removal, and for adhesive bonding. Laser ablation coating removal (LACR) is gaining acknowledgment as a potential replacement for the current method of grit blasting for surface cleaning and preparation in niche applications. In this work, the feasibility of using LACR to remove old coatings and corrosion products from carbon steel substrates to prepare surfaces for recoating is investigated. Carbon steel is common structural material used in infrastructure due to its reliable mechanical properties and low cost, but due to its tendency to corrode easily, it must be heavily coated and frequently inspected. Because of the harsh service conditions that low carbon steel components are often exposed to, coatings deteriorate and require removal followed by stringent surface preparation for recoating. Surface preparation is a key step in order to ensure that the newly reapplied coating will adhere and perform as expected over the entire service time of the coatings and meet industry standards. In order to test the effectiveness of laser cleaning for surface preparation, extensive adhesion testing of laser cleaned surfaces is needed to determine the feasibility of laser cleaning for steel surface preparation. To investigate the use of LACR to prepare surfaces, steel plates were subjected to laser ablation coating removal (LACR), induction coating removal (ICR), a combination of ICR and LACR, in addition to the incumbent cleaning method of grit blasting. The surfaces were then recoated using a typical three-layer coating system consisting of both organic (epoxy) and inorganic (silica based) zinc rich primers. Pull-off adhesion testing in addition to detailed surface analysis using scanning electron microscopy (SEM) and profilometry show that despite a lower average roughness on the LACR surfaces (avg. Ra 4 µm), the adhesion is equivalent to that of the grit blasted steel (avg. Ra 12.3 µm). In addition, complementary chemical analysis was performed using both energy dispersive spectroscopy (EDS) and x-ray photoelectron spectroscopy (XPS) on the cleaned surfaces. These results show that LACR is a valid candidate that rivals the incumbent method of grit blasting in terms of safety, cleanliness, and environmental sustainability, for both surface cleaning and preparation.

SE-ThP-5 Study of the Corrosion Resistance and Adhesion of DLC with a CrC/CrCN/Cr Bonding Multilayer Deposited by HIPIMS on AISI 4317 Steel, ***Martin Flores,*** *L. Flores,* Universidad de Guadalajara, Mexico; *J. Aguilar, A. Gonzalez,* Universidad Autonoma de Tamaulipas, Mexico

Diamond-like carbon (DLC) coatings are utilized in a wide range of applications to reduce the sliding friction, wear and corrosion resistance of components. AISI 4317 steel is used in machinery bearings for the transport of minerals with sulfur content in port facilities. These bearings suffer from wear and corrosion promoted by sulfide and chloride ions. On many substrates, DLC has limited adhesion, so it is necessary to develop interfaces and bonding layers to overcome this limitation. One way to increase adhesion and reduce corrosion is by metal ion etching using the HIPIMS technique and gradual bond layers.The metal ion etching was performed with a delay in the synchronized polarization pulse of the substrate with respect to the applied to the Cr target. This work reports the results of the potentiodynamic polarization and Daimler-Benz Rockwell C test methods used to evaluate corrosion and adhesion respectively. Synthetic seawater and seawater plus dilute sulfuric acid were used as electrolytes. Raman spectroscopy was used to study the sp2 and sp3 content of the DLC layer. SEM was used to observe the cross section and corroded surface of coated and uncoated samples of AISI 4317 steel. The structure of the bonding multilayer was studied by XRD. The results show an improvement in the corrosion resistance of the samples coated with the multilayer, the correlation between the adhesion and corrosion tests is analyzed.

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